TOBACCO BIOCHEMISTRY

2023135100

PROGRAM NUMBER

920110

PROGRAM NAME

Tobacco Biochemistry

PROGRAM COORDINATOR:

R. A. Carchman

WRITTEN BY

R. A. Carchman

PERIOD COVERED

D. Ayers, S. Hassam, R. Izac and O. Gaines Third Quarter, 1992

Coordinator Summary: Operational plans for this program were extensively reviewed after business decisions to move TSNA research to PME and suspend research on biochemical modifications to tobacco. Priority has been given to those TSNA activities which were felt most important by PME, but, due to staffing or instrumentation limitations, could not be efficiently conducted there. Research plans in the biochemical modifications to tobacco area were altered based upon advice as to which activities were most important to support current patent disclosures/applications.

- I. Objective: To determine tobacco bound precursor/smoke product relationships for MS NNK by December 1992.
 - A. Strategy: Devise methods to reduce MS NNK from bound precursors during smoking.
 - 1. Results: As reported earlier use of ¹⁴C-NNN as a recovery standard in TSNA analyses was discontinued. All TSNA data are now reported on an as is basis. The nitroso derivative of N-n-butyl-3-picolylamine (BPA) was identified earlier as a suitable recovery standard for TSNA analysis. The precursor N-butyl-3-picolylamine was prepared by alkylation of 3-picolylamine with n-butyl bromide. The reaction mixture was purified to remove the starting material and a side product, the dibutylated amine. Analysis of the N-n-butyl-3-picolylamine by GC/MSD showed it to be >99.5% pure. The N-n-butyl-3-picolylamine was nitrosated to yield N-nitroso-N-n-butyl-3-picolylamine. This product needs to be purified further before use.
 - 2. Plans: Complete the development of an appropriate recovery standard for TSNA analyses. Complete current investigations to isolate a bound form of nicotine from burley extracts. Complete current investigations to synthesize a bound form of nicotine. Study SCFE and enzymatic methods for the release of NNK from its bound form in filler. Analyze 1991 field-grown Bu 21 leaf samples for bound nicotine formation during curing.
 - 3. Contributors: S. Haut, R. Izac, R. Forte, R. Kaiser, P. Kurth, S. Hassam, W. Hempfling.

4. References:

- 1. Haut, S. Notebook No. 9078, p. 192; Notebook No. 9200, p. 14.
- 2. Forte, R. Notebook No. 9133, pp. 98, 110 and 120.
- II. Objective: To design a model cigarette with reduced MS TSNA delivery by the end of 1993.
 - A. Strategy: Reduce MS TSNA by modification of cigarette construction parameters.
 - Results: Determination of MS TSNA and TPM of 30 different codes from the cigarette construction parameter study were completed. These codes differ in amount of burley in the filler blend, paper porosity, type and level of additive, and filter ventilation and efficiency. NNN and NAT levels ranged over an order of magnitude and NNK values varied threefold. Other pertinent data, such as gas phase delivery and peak coal temperature, has also been obtained.

Determination of MS TSNA and TPM of 4 cigarette codes (nonfilter) with different circumferences has been completed (Project Wide). As was observed in a previous study, cigarette circumference has no effect on the delivery of TSNA into MS smoke. Other pertinent data, such as gas phase delivery, has also been obtained. Analysis of the data is in progress.

- 2. Plans: Evaluate data for use as a predictor for MS TSNA delivery of a given cigarette model.
- 3. Contributors: R. Izac, S. Haut, R. Forte, R. Kaiser, P. Kurth.

4. Reference:

- 1. Izac, R. Notebook No. 8874, pp. 191-199.
- 2. Forte, R. Notebook No. 9133, pp. 110 and 120.
- B. Strategy: Reduce MS TSNA by use of tobaccos naturally low in preformed TSNA, minor alkaloids and/or nitrosating agents.
 - 1. Results: Various discussions were held with other PM personnel regarding possible reasons for the lower filler and MS smoke TSNA levels in Japanese tobacco compared to U.S. tobaccos. Analyses of specific biochemical and chemical properties were initiated for a set of Japanese fillers. Chlorophyll analyses of selected Japanese burley and flue-cured fillers from a previous study showed these fillers to be comparable to DBC Bu and Br fillers.

A second set of Japanese tobaccos were analyzed for nicotine and minor alkaloids. Ranges for nicotine were 1.0 to 5.9%, ranges for nomicotine were 0.01 to 0.8%, ranges for anabasine were 0.01 to 0.04% and ranges for anabasine were 0.01 to 0.15%.

The possible presence of bound precursors (for MS NNK) was investigated by measuring MS NNK levels in one water-washed Japanese burley (BBL2NG '88). MS NNK was still present in the smoke from the water-washed filler in amounts higher than the filler NNK, suggesting that the same or similar types of bound precursors exist in this Japanese burley as are present in the DBC burleys. Similar results were also observed for a filler from the DBC Bu component study (X6DID). Nitrate levels of these samples ranged from 0.001 to 2.6%.

To acquire a better understanding of how TSNA formation is affected by cultivar type, analyses of two chlorophyll-deficient Brazilian tobacco fillers were initiated. In general, the flue-cured samples have higher preformed TSNA levels than the air-cured ones. Two anomalies were observed as well. TSNA levels for air-cured Galpao were found to be higher than flue-cured Galpao and levels for a fire-cured sample were lower than expected. Alkaloid levels were measured for these tobaccos as well. Nicotine was found to range from 11.0 to 5.1%, nornicotine from 0.01 to 0.05%, anabasine from 0.003 to 0.02% and anatabine from 0.01 to 0.06%.

- 2. Plans: Complete analyses (chlorophyll, minor alkaloids, nitrates) of Japanese and other fillers. Complete documentation of all analyses.
- 3. Contributors: W. Hempfling, S. Haut, R. Forte, R. Kaiser, P. Kurth, R. Izac, S. Hassam.

4. Reference:

- 1. Haut, S. A. Notebook 9078, p. 191; Notebook 9200, p.14.
- 2. Kurth, P. Notebook 9190, pp.
- 3. Forte, R. Notebook 9133, p. 110.
- 4. Kaiser, R. Notebook 8075, p. 94.
- III. Objective: To quantify the effect of cigarette construction parameters and specific additives on the biological activity of CSC by the end of 1992.
 - A. <u>Strategy:</u> Determine the interactive effects of modifications to conventional cigarette construction on the S/M activity of CSC.

1. Results: A set of 30 different codes of cigarettes has been sent to INBIFO. These cigarettes differ in amount of burley in the filler blend; paper porosity, type and level of additive; and filter ventilation and efficiency. All other pertinent data has been obtained.

Another set of cigarettes has been sent to INBIFO for a study to investigate the effect of cigarette circumferences on biological activity. Special inserts for their smoking machines were fabricated and sent to INBIFO.

- 2. Plans: Complete evaluation and documentation of results.
- 3. Contributors: R. Izac, INBIFO.
- 4. Reference: Izac, R. Notebook No. 8874, pp. 191-197.
- IV. Objective: Modify tobacco plants so that they produce reduced levels of alkaloids as compared to the cultivars currently in use by December, 1995 (PMT-based modification, December, 1993 and an MPO-based modification, December, 1995)
 - A. Strategy: Influence tobacco biochemistry by expressing the antisense of the cDNA sequences that are overly expressed in tobacco root to reduce the level of alkaloids in tobacco plants.
 - 1. Results: To date more than 128 transgenic plants (including controls) have been regenerated containing antisense constructs derived from the differential hybridization study. The majority of these plants have been sampled for preand post-topping alkaloid levels and most of these samples are currently being stored pending assay.

Analyses of transgenic plants for their individual alkaloid production profile has begun. An internal standard method was tested and the data handling was streamlined by the utilization of a spreadsheet (Excel).

- 2. Plans: The remaining transgenics will be assayed for pre- and post-topping alkaloid levels and the seed from these plants collected.
- 3. Contributors: Mt Shulleeta, B. Vaughan, R. Forte, ARD.
- 4. References:
 - 1. Shulleeta, M. Notebook No. 9132, pp. 43-51.
 - 2. Forte, R. Notebook No. 9133, p. 120.
- B. Strategy: Express the antisense of the DNA sequences for PMT in tobacco plants.

1. Results: The 1.2 Kb fragment of the PMT gene was cloned in the sense (1.2S) and antisense (1.2A) orientation. These plasmids were then introduced into Agrobacterium and used to transform Burley 21 leaf. Transgenic plants are presently being regenerated.

Several primers were designed to aid in the isolation of the intact PMT gene. These primers have been used in several RNA PCR experiments that are presently being analyzed.

The antibodies to the synthetic 29 amino acid peptide (N29) generated in rabbits are being used in an attempt to demonstrate the specificity of this antibody for PMT. The antibody has been used in immunoprecipitation and immunoaffinity column chromatography. In the immunoprecipitation studies the antibody is used to coat agarose beads. These coated beads were then mixed with PMT, allowed to precipitate and the PMT activity remaining in the supernatant was tested. To date no significant reduction in PMT activity has been seen. These results did not change when the order of the reagents were changed. This antibody has also been used in an affinity column. The column did not adsorb the expected protein from solution under the conditions tested.

None of the plaques from the tobacco root cDNA library screened with antibody obtained from the 60 Kd protein were identified as unambiguously positive. This line of work has therefore been abandoned until other antibodies become available.

- 2. Plans: Continue to use PCR technology and the DNA sequence data to isolate the intact putative PMT gene. Complete the sequencing of the intact putative PMT gene. Attempt to express the putative PMT (60 kD polypeptide) gene in E. coli. Continue plans to demonstrate the specificity of the antibody for PMT using different conditions for immunoprecipitation and affinity chromoagraphy.
- 3. Contributors: M. Krauss, T. Michalik, M. Shulleeta, B. Vaughan, S. Wahab.

4. References:

- 1. Krauss, M. Notebook No. 9156, pp. 174-200 and 9204, pp. 1-13.
- 2. Shulleeta, M. Notebook No. 9132, pp. 43-51.
- 3. Vaughan, B. Notebook No. 9206, p. 8.
- 4. Wahab, S. Notebook No. 9201, pp. 70-73.
- C. Strategy: Reduce the level of nicotine by affecting the biosynthetic pathway at nicotine synthase.

1. Results: Crude [2,6-¹⁴C]quinoline, prepared from radiolabeled glycerol and aniline, was purified by HPLC. The quinoline was oxidized with selenium in sulfuric acid to nicotinic acid, which was purified by sublimation. By this method, about 30 μCi of ¹⁴C-nicotinic acid was prepared, with a radiochemical purity of 99% and a chemical purity of about 98%.

The commercially available nicotine ELISA was used to quantitate nicotine synthase activity in tobacco root extracts required by the Tobacco Biochemistry Program. A set of five samples were tested in the nicotine ELISA. The assay detected less than 1 ppb nicotine in three of the control samples tested - dibasic sodium phosphate, nicotinic acid and enzyme preparation. The concentration of nicotine detected in two samples - a combination of buffer, nicotinic acid, N-methylpyrrolinium and PD-10 purified enzyme; and a combination of buffer, nicotinic acid, N-methylpyrrolinium, and non-purified enzyme - was 5.4 and 102 ppb, respectively. Relative to their respective controls, no increase in nicotine was detected in either the crude or partially purified enzymatic preparations. However the data showed that PD-10 purification reduced the endogenous level of nicotine prior to experiments designed to measure the activity of nicotine synthase.

- 2. Plans: No further quantitation of nicotine synthase activity is planned at this time due to a restructuring of the Tobacco Biochemistry Program.
- 3. Contributors: R. Izac, M. Steele, E. Morrisette.

4. Reference:

- 1. Izac, R. Notebook No. 8874, pp. 191-197.
- 2. Steele, M. Notebook No. 9068, pp. 194-195.
- 3. Morrisette, E. Notebook No. 9176, pp. 78-86.

ENVIRONMENTAL SUPPÖRT

2023135107

Source: https://www.industrydocuments.ucsf.edu/docs/ltjl0000

PROGRAM NUMBER : 920111

PROGRAM NAME : Environmental Support

PROGRAM COORDINATOR: C. R. Hayward
PERIOD COVERED: Third Quarter, 1992

Coordinator Summary: The use of water scrubbing has been shown to be effective in removal of propylene glycol and nicotine as predicted. The operation of the pilot scrubber continues to provide quantities of scrubber water to demonstrate the use of bioremediation process to eliminate materials scrubbed from the stack gases.

Laboratory data shows that nicotine can be removed from the combined sludges at Park 500 using aeration. A scale up in size is planned to confirm these results.

Efforts continue to support the stack gas evaluation program, the phosphine control developments, and the potassium nitrate quality improvement.

- I. Objective: Develop technology to reduce the levels of various components (nicotine, ammonia, and VOC's) in air emissions at the BL Plant to support Environmental Engineering in maintaining compliance at the site and preparing for future permitting requirements.
 - A. <u>Strategy</u>: Evaluate adsorption technology for either direct treatment of air streams or for treating blowdown from a scrubber.
 - 1. Results: As reported last quarter, direct treatment of the air streams with an adsorbant was abandoned due to excessive pressure drops encountered using XAD- 4 resin.

Additional studies were conducted using XAD-4 to treat water from the pilot scrubber to remove nicotine, with the assumption being that the treated water from a full-scale system, containing PG and ammonia, could then be discharged to the city sewer system. The adsorption tests were quite successful, with nicotine loadings on the resin of up to 10% by weight.

Based on work conducted by Chemical Research, regeneration tests were run using 6% citric acid to pull the nicotine back off of the resin. These tests were successful in producing a relatively concentrated regenerant (1-2% nicotine). However, the XAD-4 retained a slight discoloration, and analysis of a secondary ethanol regeneration showed that there were adsorbed compounds that were not being removed by the citric acid.

At this point, preliminary discussions with the city indicated that the ammonia level in the scrubber wastewater was too high to discharge directly to the sewer. Since XAD-4 does not remove ammonia, this meant that a second treatment step would be required for ammonia reduction prior to sewer discharge. At this point, it was decided that a biological treatment process would be preferable to an adsorption process.

- 2. Plans: No further tests are planned for the immediate future. Should scrubbing be implemented at the BL Plant, the concept of recycling the scrubber water after nicotine removal with XAD-4 will be evaluate.
- 3. <u>Conclusions:</u> XAD-4 remains an effective adsorbent for removing nicotine from aqueous solutions. Questions remain about the optimum regeneration process, however.
- 4. Contributors: Chemical Research Division; Reconstituted Tobacco
- B. Strategy: Evaluate scrubbing technology for treating the air discharges.
 - 1. Results: Tests with the pilot scrubber at the BL Plant were completed. The 300 cfm stream from the Line 2 south main dryer exhaust was effectively scrubbed with water flows as low as 5 gallons per hour. At this water rate, 97% of the PG and 80% of the nicotine was removed.

Using these conditions as a basis, a preliminary design was developed for the BL Plant in which a scrubber would be installed on each of the three drying lines, with each scrubber treating about 60,000 ACFM. The blowdown rate for all three scrubbers would total about 50 GPM.

Based on the air volume to be treated, the preliminary design calls for 13- foot diameter Sly Impinjet scrubbers. Arrangements have been made to test using Sly's pilot scrubbing system to verify the design and determine the number of trays required. This scrubber should be available in late October or early November.

A survey was conducted by McNamee Advanced Technology of various treatment alternatives for the scrubber wastewater. After elimination of the adsorption alternatives, it was determined that a biological treatment step would be the most cost-effective and reliable means of treating the water.

An anaerobic process, known as an Upflow Anaerobic Sludge Blanket (UASB), was investigated by Biothan, Inc., in New Jersey, using wastewater from the pilot scrubber. This system was very effective in removing BOD from the water, but analysis of samples sent to us by Biothane showed that there was no degradation of the nicotine. Based on these results, the UASB concept was discarded!

The recommended treatment in an aerobic activated sludge process using a sequential batch reactor. This process will degrade all three components - PG, nicotine, and ammonia - in the wastewater. A preliminary design was obtained from Aqua-Aerobics, and pilot testing has started to verify their design.

PM Engineering is preparing a cost estimate for the scrubbing and water treatment system for comparison with the incineration alternative.

- 2. Plans: The pilot packed scrubber at the BL Plant will continue to operate to provide wastewater feed for the pilot sequential batch reactor. The Sly unit will be installed and evaluated when it is available.
- 3. Conclusions: Water scrubbing is: an attractive alternative for treating the BL Plant air discharges, and an aerobic activated sludge water treatment process is the only viable alternative of the wastewater.
- 4. Contributors: Reconstituted Tobacco Development
- II. Objective: Develop alternatives for the current discharge of burley stem washing extract into the Richmond sewer system.
 - A. Strategy: Evaluate elimination of the burley stem washing process for RCB.
 - 1. Results: Flavor Technology is coordinating the evaluation of the subjectives.
 - 2. Plans: Subjective testing will continue prior to implementing the process modifications in the BL Plant.
 - 3. Contributors: Reconstituted Tobacco Development, Flavor Technology Division, Product Evaluation Division
- III. Objective: Develop a plan to reduce or eliminate potential environmental and disposal problems associated with the potassium nitrate co-product from the RL denitration process.
 - A. <u>Strategy</u>: Evaluate purification processes for the potassium nitrate that will improve the quality of this product.
 - 1. Results and Conclusions: A number of alternate processes for using XAD-4 resin to adsorb impurities are under evaluation. XAD-4 resin has been demonstrated as being the most effective adsorber of impurities from the potassium nitrate solution.
 - 2. Plans: Continue to evaluate process alternates and select the best method to begin laboratory development.
 - 3. Contributors: Chemical Research Division, Reconstituted Tobacco
 Development
- IV. Objective: Provide support to the R&D and Engineering Five Year Plans addressing air, water, and solid waste issues.
 - A. Strategy: Provide analytical expertise related to sample protocols for qualitative and quantitative descriptions of air, water, and solid waste effluents.

- 1. Results: Support to Engineering was provided to in qualifying and auditing outside laboratories, developing requested physical data, providing updated laboratory procedures, and support development of solid waste volume reduction process.
- Plans: Continue support.
- 3. Contributors: Analytical Research, Chemical Research
- B. <u>Strategy</u>: Provide sampling and analytical protocols in support of studies related to Project Grain.
 - 1. Results: Support of analysis and protocols for ethanol and humectants in stack gases continued.
 - 2. Plans: Continue support.
 - 3. Contributors: Analytical Research
- C. <u>Strategy</u>: Support the development of improved methods for reducing the phosphine levels from warehouse fumigation.
 - 1. Results: Determined the catalyst levels on the carbon prior to use and after regeneration. Evaluated the TCLP results of the "spent carbon" and found it to be a non-hazardous waste.
 - 2. Plans: Continue support of Engineering Development plans.
 - 3. Contributors: Chemical Research, Analytical Research
- V. <u>Objective</u>: To evaluate the use of microorganisms to reduce the levels of selected compounds in different processes.
 - A. <u>Strategy</u>: Determine the bioremediation conditions required to remove tobacco alkaloids (TA) from the centrifuge sludge (CS) coming from the Park 500 process:
 - 1. Results: CS, press cake (PC) and KNO₃ were mixed at the ratio of 1:2:0.05. In shake flask studies, it was shown that TA was reduced in the mixture at a faster rate than with the three components alone. The TA removal rate occurred at a faster rate in the mixture with adequate aeration and at temperatures between 4°-37°C than in an unaerated mixture over the same temperature range.

In box studies, TA reduction was best with a CS:PC:KNO₃ mixture (1:2:0:05) with daily stirring. CS alone did not show significant TA reduction after 45 days of incubation at room temperature.

- 2. Plans: Study the TA removal in CS-PC mixture in a larger scale 4th Quarter 1992.
- 3. <u>Conclusions</u>: TA in a CS-PC-KNO₃ mixture can be removed to nondetectable levels with proper aeration.
- 4. Contributors: Biochemical Research Division/Project 1902

NEW PRIMARY PROCESS

2023135113

PROGRAM NUMBER :

PROGRAM NAME : New Primary Process

PROGRAM COORDINATOR: S. E. Clark
PREPARED BY C. A. Wood

PERIOD COVERED : Third Quarter, 1992

Coordinator Summary: The NPP appropriation for \$3.7 million was approved and will permit purchase, modification and installation of the equipment necessary to complete the process definition objectives for the program.

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The program is on target to deliver the design basis for a new facility. Additional emphasis is being placed on implementing technologies, developed within the program, into existing facilities where immediate benefit can be achieved. Steam tunnel treatment before aftercut drying of the BRICA total blend is the initial concept being evaluated. Unit Operations testing is continuing in the areas of microwave heating prior to cutting for use in the Primary and NET processes. Cutting and drying screening tests were completed and additional filler blending and flavor investigations continued.

New procedures are being finalized for single puff cigarette subjective analysis that will provide the basis for evaluating process influences on taste. No evidence has been seen of any changes in mobility or crystallinity in pectin or cellulose in Bright or Burley tobaccos processed through a steam tunnel and rotary dryer.

The models of an existing and a new primary were completed and will be used for evaluating the design basis for a new facility as well as implementation scenarios for NPP technology in existing facilities.

- I. Objective: Develop a process for preparing blend components individually or in selected combinations which manages the interactive objectives of maximizing the cigarettes produced per pound of tobacco utilized and achieving subjective acceptability of the blend.
 - A. Strategy: Utilizing various blending techniques, demonstrate that individually processed components can be blended to a degree of uniformity that is at least comparable to current total blend process methods.
 - 1. Results: Previous studies utilizing natural chemical markers found no blend differences between cigarettes produced via individual component processing (ICP) and the conventional strip blending process. The latest investigation evaluated variations in rod weight, RTD, and static burn. Cigarettes produced via ICP had higher variation in RTD. Picking analysis of the cigarettes found no indication of differences in blend uniformity. The ICP cigarettes contained significantly longer filler (4.8% vs 3.7% 6 mesh) and may be the cause of the RTD variation since the picking data suggests no blend difference.

It was previously reported that a modified Rothmans three wheel carding device was effective at declumping 100% cut RL in preliminary testing, producing a

uniform, well separated discharge stream. In additional testing, the unit also provided a significant amount of in-line blending of the individual blend components. Based on these results, an increased capacity unit for in-line blending was designed for incorporation into the NPP related Semiworks modifications.

- 2. Plans: The existing three wheel carding unit will be incorporated into the modified Semiworks addback area to declump 100% cut RL prior to blending with the remaining blend components. An increased capacity, six wheel unit will be procured and installed to provide in-line blending of the combined components. Semiworks modifications to permit simultaneous feed of all six blend components will be accomplished in the fourth quarter.
- 3. Conclusions: Direct measures of blend uniformity picking and chemical markers have provided no indication of blending differences between individual component processing and conventional strip processing. Secondary measures have shown increased variability with ICP, however, such measures are also adversely affected by the increased filler size which is obtained with ICP. It can be hypothesized that the increased variability of the secondary measures is attributable to filler size increases rather than blending differences. Additional data will be collected to investigate the blending efficiency of the new Semiworks cut component blending line in the fourth quarter.
- 4. Contributors: J. Gear, B. Handy, D. Lisbon, R. Mullins
- 5. References: R. Mullins, Presentation, "Blending Investigations, NPP," February 18, 1992.

James Gear, Memo, "Sampling Plan for Comparing Different Models of Blending Processes," November 6, 1991.

- R. Mullins, Memo, "Test Plan, Blending Investigation with a Simplified Blend," February 26, 1992.
- C. Wood, Presentation, "Cut Component Blending, Semiworks Tests," December 1991.
- C. Wood to R. Mullins, Memo, "Blending Technology Search," January 15, 1992.
- B. Strategy: Identify by component, specific operational parameters which provide improved strand length and cigarette firmness. Set priorities for unit operation activities based on their potential impact on cut component physical quality performance.
 - 1. Results: A report covering Hauni Tunnel treatment of individually processed components was issued.

Several tests were completed which demonstrated that Hauni Tunnel treatment of the BRICA blend allows cigarette weight to be reduced by as much as 35 mg relative: to an untreated control model at the same firmness. Alternate implementation scenarios and schedules for introducing the technology into Production are currently being developed in cooperation with Engineering, Production Planning, and Manufacturing.

Additional testing to determine the impact of either cooling filler or applying aftercut prior to the addition of expanded components failed to find any improvement in either filler CV or cigarette firmness.

An experimental test matrix of 28 tests was designed and completed which permitted a two level screening evaluation of each of eight operating parameters, and their interactions, for cutting and drying uncased E33 Bright tobacco. A full factorial test matrix would have consisted of 192 tests. The test matrix did not involve cutting at low moisture as the necessary equipment was not available. The parameter found to be most significant in influencing CV was treatment of the filler in the Hauni tunnel after cutting. An average 0.4 cc/gm improvement in CV was achieved with tunnel treatment. The other factors which were found to significantly influence CV and shred size were: cutting OV, cutting width, tunnel residence time, and dryer residence time. 21% cutting OV was preferable to 27%, 17.5 cuts/inch was preferable to 35 cuts/inch, 20 sec. tunnel residence time was preferable to 70 sec., and 3 minutes dryer residence was preferable to 7 minutes.

2. Plans: Define cigarette design changes needed to acceptably match delivery, puff count, and subjective targets of BRICA products at reduced weight. Evaluate the various implementation scenarios for steaming tunnels and recommend the most appropriate one for effectively transferring the technology to Manufacturing by October 1992.

Since two investigations have failed to find any improvement in cigarette firmness attributable to cooling filler or applying aftercut prior to the addition of expanded components, no further testing of these process modifications is planned.

Using the results obtained from the screening tests for E33 Bright, a test matrix has been designed to determine the operating parameter settings for the present Semiworks configuration which yield maximum filling value for each component. The assumption has been made that those parameters which were found to most influence CV and shred size for Bright will also have the most influence on the other components. The test matrix consists of 15 tests per component, and began the week of August 24, 1992.

3. Conclusions: BRICA based cigarette production is projected to be 41.4 billion units in 1993, full implementation of this technology in Manufacturing offers a potential savings of \$5.8 million/year. An aggressive implementation plan is warranted due to this cost savings potential.

The factor found to have the most effect on the filling value of E33 Bright during cutting and drying was use of the Hauni tunnel. Treatment of the filler in the tunnel after cutting provides the opportunity for increasing CV by at least an average 0.4 cc/gm. Optimization of residence time, tobacco bed depth and steam/tobacco ratio in the tunnel may increase this improvement. Other parameters found to be important for optimizing filling power were: cutting OV, cutting width and dryer residence time.

- 4. Contributors: J. Crump, J. Gear, J. Nepomuceno
- 5. References: R. S. Mullins and D. P. Lisbon, R&D Special Report 92-018, "Impact of Hauni Tunnel Treatment on the Physical Properties of Individual Processed Components," R&D Special Report 92-018, June 12, 1992.
- C. <u>Strategy</u>: Conduct laboratory tests directed toward identifying and obtaining a better understanding of the mechanisms which influence filling power.
 - 1. Results: ¹³C CPMAS and ¹H MAS NMR have been employed to detect chemical and physical changes in tobacco lamina resulting from various processing regimens involving treatment of tobacco with the Hauni steam tunnel and KLK dryer.

In uncased Bright tobacco, the ¹³C resonances of a neutral sugar carbohydrate component were found to increase following tunnel and dryer treatment when both the before and after treatment samples were studied after equilibrating to about 10% OV. This increase is interpreted as resulting from an increase in the rate of polarization transfer in the ¹³C CPMAS experiment which may be due to a restriction of the rotational mobility of the carbohydrate carbons following heat treatment, or an increase in the amount of immobilized water in the carbohydrate. This effect may be related to the tobacco stiffening since it is known that low frequency motions are correlated to mechanical properties of polymers. CV values for the samples before and after tunnel drying were 3.91 cc/gm and 4.92 cc/gm. Enhancement of the same carbohydrate resonances was not observed for samples when they were equilibrated at about 14% OV. It is not known why the effect disappears under these conditions. It is likely that the differential effect becomes much smaller at higher OVs making it more difficult to detect.

In cased Burley tobacco, the resonances of citrate increased while those of fructose disappeared following tunnel and dryer processing. No other changes were observed. The increase in citrate may be due to an actual increase in citrate (from oxidation of fructose, for example) on it may be an apparent increase because of changes in line widths due to the crystallization of dispersed citrate. The latter may be promoted by the migration of calcium ions during the hydration of the tobacco.

NMR results obtained for oriental tobacco are inconclusive due to differences in the moisture content of the samples tested.

No evidence was observed by NMR for any change in mobility, ordering, or crystallinity in either pectin or cellulose in Bright or Burley tobacco after processing through the steam tunnel and dryer. In addition, extraction-dialysis-freeze drying also failed to show any changes in the amount of free pectin in Bright tobacco after treatment in the Hauni tunnel and subsequent drying.

Stress-strain tests were completed on Bright lamina samples at 65% RH, 60% RH, 50% RH, and 40% RH and at temperatures of 23.5°C, 40°C, 50°C, 60°C, and 70°C. At the lower relative humidities (i.e. 50% RH and 40% RH), the effect of moisture was decreased so that the dominant effect due to temperature change could be determined.

The breakage properties of lamina showed a transition similar to the glass transition between 60°C and 70°C at both 40% RH and 50% RH. The toughness and the strain-to-break showed an approximate two fold increase in this region. This change is significant considering that the deviation from 23.5°C to 60°C was relatively small and that the wide range of polymers in tobacco lamina would tend to broaden such effects. At 65% RH and 60% RH, the effect of temperature was less than that at the lower humidities because of the increased moisture content. The plasticizing effect of water on the Bright lamina even at 40% RH is still enough that it will decrease the glass transition temperature and broaden the transition region.

The lamina breakage characteristics at 70°C and 50% RH (10.5% OV) are similar to those obtained at 40°C and 60% RH (13% OV). Furthermore, no transition is observed below 60°C independent of the humidity at which the sample is equilibrated.

2. Plans: NMR studies will continue to clarify the effect that sample moisture content has on the observed results and to establish whether the observations can be directly correlated to filling value differences.

Testing will begin to develop a method for testing the mechanical properties of cut filler. Initial tests will be with RL sheet cut with a paper cutter to minimize sample to sample nonuniformity. Tension and bending tests will be performed on individual shreds. In addition, stress relaxation measurements will be made on a volume of cut shreds using a method similar to the Borgwaldt CV testing method. These tests will permit evaluation of the effects of processing mechanical properties affecting filling value.

3. Conclusions: No evidence has been seen using either NMR or extraction-dialysis-freeze drying methods for any change in mobility, ordering, or crystallinity in pectin or cellulose in Bright or Burley after processing through the Hauni tunnel and KLK dryer.

NMR studies have shown differences in the ¹³C resonances of a neutral sugar carbohydrate component for uncased Bright tobacco following tunnel-dryer treatment. For cased Burley, an increase in citrate resonances and decrease in fructose resonances were observed after tunnel-dryer processing. However, whether these differences are observed apparently depends on the moisture content of the sample, and further testing is necessary before a conclusion can be made as to whether any correlation exists with differences in filling values.

- 4. Contributors: A. Basak, G. Chan, J. Crump, W. B. Edwards, S. Ganeriwala, J. Wooten
- D. <u>Strategy:</u> Evaluate the feasibility of applying microwave heating to tobacco strip prior to cutting at reduced moisture levels.
 - 1. Results: Bright, Burley, RL 150B and RCB strip at 13% OV were cut via the Legg Millicutter at the Semiworks after heating the strip in a batch microwave unit to approximately 150°F to 160°F. These samples were subsequently ordered from 13% to 16% followed by air drying back to 13% OV. CV results for these tests were equivalent to CV values obtained from control samples cut at 21% then dried to 13%.

A large quantity of the NET Bright blend (#10 Bright cased with a sugar casing) was heated with the microwave at 16% and cut at this low moisture content using the Millicutter. This material was then expanded through the NET process in the 8" tower (Phase III). Due to the higher bulk density of this material, the impregnator capacity increased from 225 to 380 pounds per impregnator load. Other than the proportionately longer cooling step, no problems were encountered with this higher loading through the impregnation cycle. Uniform bed temperatures (-8 to 0°F) were measured within the bed at the end of the impregnation cycle. Tower gas temperatures of 520, 550 and 580°F were used for the expansion step. CV and sieve results for these tests will be available within the next week.

Modifications to the microwave heating/cutting process are currently in progress at the Semiworks, with the work scheduled for completion by the second week of September. These modifications will allow processing of tobacco in a continuous mode for the evaluation of various techniques to enhance the filling power for cut components after microwave heating and cutting.

2. Plans: Complete modifications to the microwave/cutting process at the Semiworks and begin evaluation of various process techniques to enhance the filling power of cut components after microwave heating and cutting. A proposal will be solicited for a commercial-scale microwave unit suitable for the NET process.

- 3. Conclusions: Microwave heating and cutting of NET feedstock increases the capacity of the Phase III impregnator by approximately 65% to 70%. The potential for eliminating the conventional drying step for NET infeed material will be evaluated based on CV results from these tests.
- 4. Contributors: P. Chen, T. W. Howell, J. Nepomuceno
- E. Strategy: Develop a database to record processing parameters and to provide access to analytical data for New Primary samples.
 - 1. Results: All forms that were developed by Pantajja Consulting have been received and tested. The remainder of the work is creation of several end-user reports and access programs. These will be completed by mid-September.
 - 2. <u>Plans:</u> Work with users and make changes and corrections in order to complete original project design specifications.
 - 3. Contributors: B. Good, R. Lipps
 - 4. References: B. Good to Distribution, Memo, August 21, 1992.
 - B. Good to C. Rowe, Memo, "New Primary Process Database Development," January 27, 1992.
 - R. N. Lipps to Distribution, Memo, "New Primary Process Database Implementation," April 21, 1992.
- II. Objective: Develop flavoring systems that compliment the process developed in this program and achieve the subjective acceptability necessary to produce existing and additional value added products.
 - A. Strategy: Develop casing/flavor systems to achieve subjective parity between individual cut component processing and total blend processing.

Formulate casing/flavor systems to achieve lower costs and reduced thermal treatment through consolidation of unit operations.

Consolidate casing/flavor systems to reduce cut component storage needs.

1. Results: Baseline responses have been established on two models (Marlboro and Bristol) produced with the NPP processing scenario using the existing flavor systems. Sensory differences were discerned between the test and control models. This difference is suspected to be due to a lack of flavor transfer from the cased components to the reconstituted components. Initial testing has indicated a significant transfer of certain casing components to the reconstituted portion during Total Blend processing, primarily during the cutting operation.

A flavor system has been installed in the DCC to combine the conditioning and casing operations. The unit operation will eliminate the thermal treatment, moisture gains, and capital costs associated with separate casing. An evaluation of this system for subjective parity with the traditional method is in progress.

Models aimed at lowering the OV gain associated with Burley Spray through reformulation have been incorporated into the BRICA formulation. A DCC flavor application of a reallocated portion of the Burley Spray to lower the moisture gains is being evaluated.

A study was conducted to determine the survivability and subjective impact of reducing/eliminating PG and glycerin from casings. Samples are currently being evaluated.

Casing rate consolidation studies have not commenced.

2. Plans: Develop casings for reconstituted tobacco to compensate for the lack of transfer normally occurring the total blend cutting operation.

Qualify the DCC as an alternative method of casing. Incorporate post-cutting application of casings on the Burley portion to eliminate the separate casing and drying operations.

Complete the evaluation of reduction/elimination of humectants in casings.

- 3. Conclusions: Lack of casing transfers appears to a potential reason for the sensory differences noted with individual cut component processing.
- 4. Contributors: B. Bell, D. Ennis, H. Spielberg, D. Rockwell, R. Pitts
- B. Strategy: To provide expert testing and consumer testing of cigarette models resulting from the development of the New Primary Process (NPP) at various stages in its development. To establish the sensory basis for qualifying the NPP as a viable alternative to the current primary process and to develop an understanding of the effect of unit operation variables on cigarette sensory parameters.
 - 1. Results: Procedures for single puff smoking of cigarettes were developed further. Conventional machine smoking of cigarettes was replaced with a new approach based on a manifold vacuum system. This equipment was initially tested in T4 Conference Room for several weeks before being permanently installed in the Panel Room on A1. Work on sequence effects, rinsing, feedback and interpuff intervals was conducted using two types of Bristol cigarettes and a Bright and Bright/Oriental blend. Individual differences were also studied in which discriminal distance (d') and bias (β) were evaluated for 1.1 panelists. These results were presented to the NPP team. There were large individual differences between subjects in their ability to detect differences between the cigarettes tested, but response bias was, in general, not fan from that of an ideal observer.

- 2. Plans: In order to overcome some problems with sequence effects and with training on a variable product like a cigarette, research using "same-different" judgments will be initiated. To use this method, subjects need no training and it may be more suitable to the detection of small differences in a noisy product like a cigarette. This theory can be used to develop estimates of sensory differences; response biases and sensory maps of multiple products. Comparisons between this method and the identification method will be made using the BRICA blend with different steam/tobacco ratios. Following this work, sensory evaluation of individual components processed at different levels of steam, residence time and bed depth in the tunnel will be initiated.
- 3. Contributors: D. Ennis, B. Joyner, C. Hayes
- C. <u>Strategy</u>: To provide analytical data that will support the optimization of Physicals, Control Taste, and Enhance Flavor Reallocation in the development of New Primary Processing Technologies.
 - 1. Results: A Flavor Transfer Test to determine what flavors transfer during the Total Blending Process were compared to analytical results from the Individual Blending Process using RLTC, Bright, Burley, and Oriental. Samples of picked RLTC and picked Bright, Burley, and Oriental from Total Blend were analyzed for Reducing Sugars, Alkaloids, Glycyrrhizic Acid, Nitrate Nitrogen, Soluble Ammonia, Sorbic Acid, Glucose, Fructose, Sucrose, and OV. Individual samples of RLTC, Bright, Burley, and Oriental from Individual Blending Process were analyzed for the above tests.

Cigarette samples from the Bristol Blend run at the Semiworks were analyzed for Alkaloids, Total Reducing Sugars, Glucose, Fructose, Sucrose, OV, and Soluble Ammonia.

Generic Licorice Removal Study Cigarette Samples were analyzed for Alkaloids, Glucose, Fructose, Sucrose, Total Reducing Sugars, β - Methylvaleric Acid, Glycerine, Phosphorus, Soluble Ammonia, and OV.

- 2. Plans: Continue to establish Sampling Protocols which will better maintain sample integrity prior to delivery to the Chemical Analysis Section for analysis. To continue to support New Primary Processing Concepts and Design associated with Optimization of Physicals, Controlling Taste, and Flavor Reallocation.
- 3. Contributors: N. Adams, L. Branch, C. Callicutt, A. Ganzert, R. Jones, S. Langley, T. Larus, M. Mangrum, R. McDaniel, B. Ryan, D. Self, I. Smetena, K. Torrence
- III. Objective: Utilizing the NPP process, provide filler for cigarette manufacturing at a cost which is independent of the blend and quantity of filler required and can be delivered to any maker.

- A. Strategy: Develop computer models of New Primary and Existing Primary Processes which can be used to optimize the logistics of our Primary/Manufacturing operations.
 - 1. Results: Two simulation models were developed for the Cabarrus expansion based on New Primary and Existing Primary process designs. The NPP model was extended to include mobile storage and delivery systems (skips) for single blend components and cut filler, and modules in cigarette manufacturing. Extensions to the Existing Primary Model included cut filler silos and cigarette manufacturing modules.

The NPP and Existing Primary models were run using schedules of increasingly complex brand mixes along with smaller production order sizes. The NPP model results showed run time reductions and less in-progress tobacco inventory, along with less sensitivity with increasing brand complexity.

2. Plans: The Existing Primary model (Cabarrus expansion) will be converted to a model of the current Cabarrus facility. The model will be validated against current production, which requires in-depth interviews with production experts and collection of empirical production data. The complex plant-wide model will be divided into sub-models as necessary to improve simulation speed and accuracy.

The Cabarrus model will be applied to testing complex brand mixes, to assessing the benefits of separable components of NPP, and to analyzing opportunities for improving scheduling and inventory management.

Modeling efforts will be coordinated with Supply Chain on scheduling optimization and CAD on expert systems development.

- 3. Conclusions: Simulation models are an essential and powerful tool in understanding and analyzing the complex issues involved in comparing the New Primary and Existing Primary Processes.
- 4. Contributors: L. Haws, D. Kohman
- 5. References: L. A. Haws, Presentation, "Flexibility in New and Existing Primary Processes," April 10, 1992.
 - L. A. Haws, Memo, "Primary Process Simulation Operating Rules," July 10, 1992.
 - L. A. Haws, Presentation, "Process Simulation Design and Performance of New and Existing Primary Processes," July 27, 1992.
 - D. Kohman, Presentation, "Strategies and Plans: for Developing; a Cabarrus Simulation Model," July 18, 1992.

SENSORY TECHNOLOGY

2023135124

PROGRAM NUMBER : 920113

PROGRAM NAME : Sensory Technology-PROGRAM COORDINATOR: R. A. Carchman

WRITTEN BY : C. Hayes, W. Reininghaus, P. Lipowicz, J. Garman, G. Patron

PERIOD COVERED : Third Quarter, 1992

Coordinator Summary: There has been some interruption in research to allow for the relocation of the electrophysiology laboratory to INBIFO. The basification studies have been complicated by an interaction of calcium hydoxide with the casing formulation which resulted in a filler for which the pH was lower than desired and unstable. A possible solution to this is under investigation.

I. Objective: To develop the technology to produce low alkaloid and low tar/low alkaloid cigarettes that have sensory characteristics superior to currently available products.

- A. Strategy: Develop a fundamental understanding of how nicotine affects sensory systems.
 - 1. Results: A concentration response curve for (S)-nicotine has been determined by INBIFO for the olfactory epithelium of the frog. Additionally, INBIFO has established an analytical method (photoionization) for the real time online determination of nicotine vapor concentrations.
 - 2. Plans: Compare the sensory characteristics of (R)-nicotine and selected nicotine analogues to those of (S)-nicotine in the frog model. Characterize peripheral nicotine receptors using known acetylcholine agonists/ antagonists.
 - 3. Contributors: INBIFO.
- B. Strategy: Develop an analytical approach to the measurement of the acid/base character of filler and smoke.
 - 1. Results: Annular denuder and collection on XAD resin experiments were employed to estimate the vapor phase nicotine of three cigarette models ranging in filler pH from 5.2 to 8.5 (1). No differences in estimates of vapor phase nicotine were observed among the three models using the denuder method. However, a systematic relationship was observed between filler pH and nicotine trapped on the resin employing the XAD method. That is, as pH increased, more nicotine was trapped in the XAD cartridge (2).

Experiments were initiated to evaluate the stability of the pH of Ca(OH)₂ treated filler over time. Several small scale (200 grams) filler basification experiments employing the original Ca(OH)₂ casing formulation were not successful in achieving a target filler pH level of 8.0. The experimental results indicated that

the combination of Ca(OH)₂ with reducing sugars in the casing formulation does not yield a stable filler of the desired pH. Promising results have been obtained using sucrose, a non-reducing sugar, with Ca(OH)₂. Further investigation of these results is currently being conducted by the Flavor Technology Division.

The pH in smoke instrumentation, consistent with historical literature, is currently operational. Additional experimentation is underway to evaluate different buffer media, to compare whole versus gas phase smoke, and to utilize an aerosol generator.

- 2. Plans: Complete additional puff-by-puff smoke pH experimentation. Continue to evaluate the stability of the pH of basified fillers over time. Results of analytical testing will continue to be compared with subjective and evoked potential results to evaluate interrelationships that might be used to predict results and indicate directions for future models.
- 3. Contributors: CRD, BCR, ARD, CTSD, FTD.

4. References:

- Analytical Research Division. General Analytical Service Request and Data Sheet T92014, 1992 June 8.
- 2. Pierotti, J. Notebook No. 9193, pp. 2-3.
- C. Strategy: Evaluate the effects of increasing filler and smoke "pH" on the sensory and subjective characteristics of cigarettes.
 - 1. Results: Efforts are currently directed at moving the electrophysiological lab to INBIFO.
 - 2. Plans: Reestablish experimental equipment at INBIFO. Develop a nicotine/EP curve employing cigarettes at constant tar and varying nicotine levels. Conduct subjective and sensory evoked potential evaluations of cigarettes prepared from filler treated with Ca(OH)₂ and alternate bases.
 - 3. Contributors: Project 1620.
- D. Strategy: Investigate variables which affect cigarette smoke particle size.
 - 1. Results: A report entitled "Effect of Cigarette Construction and Other Variables on the Particle Size of Mainstream Smoke Aerosol" has been completed by P. J. Lipowicz and T. T. Nguyen and is currently under review in the Physical Research Division. The conclusions of the report are summarized in the Conclusions section below. These conclusions were presented at a Sensory Technology meeting on 19 August 1992.

- 2. Plans: A report which gives a theoretical basis for these findings will be completed in the fourth quarter.
- 3. Conclusions: Particle size shows little or no correlation with tobacco type, filler modification, filter, paper, or puff number. Particle size was positively correlated with butt length and ventilation, and negatively correlated with puff volume. The maximum change in particle size measured for any one of the variables was about 30%.
- 4. Contributors: Project 2704.

5. Reference:

Effect of Cigarette Construction and Other Variables on the Particle Size of Mainstream Smoke Aerosoll P. J. Lipowicz and T. T. Nguyen (in review).

- E. <u>Strategy</u>: To evaluate the applicability of filter technology to improve the sensory characteristics of low tar/low alkaloid cigarettes.
 - 1. Results: Because of the high priority placed on the De-Nic menthol program, non-menthol modeling for the Half-Nic cigarettes was temporarily deferred.
 - 2. Plans: Continue the filter/cigarette modeling when project is reactivated.
 - 3. Contributors: Project 2108.

OTHER PROGRAMS

2023135128

PROGRAM TITLE: Summary - Other Programs

WRITTEN BY : P. N. Gauvin

PERIOD COVERED: Third Quarter, 1992

The Third Quarter review on ongoing efforts in Biochemical Special Investigations indicates that experiments are continuing to develop Biosensor Technology in-house to determine the concentration of gas phase compounds. Testing was being conducted to develop/implement a nicotine ELISA for the determination of nicotine concentration. Following review of the data, no further work is planned in this area due to a restructuring of the Tobacco Biochemistry Program. Work has also been discontinued on exploring the possibility of applying the Electronic Nose developed at the University of Warwick to sensory and analytical questions currently being addressed in Philip Morris R&D. A tin oxide sensor will be developed in order to conduct similar studies in-house.

In the area of Computing Systems, the HP1000 system in Semiworks has been replaced with a Dec Micro Vax system. The TPM work station upgrade in Cigarette Testing and the automation of the computations/reporting for the Tracon x-ray have been completed.

Phase III of the Dec System 5900 installation/transition plan (to provide vendor and system software suite comparable to the existing Vax/8650 systems) will be completed by August 31, 1992, and the Vax system will be shutdown. In addition, several types of multivariate calibration techniques have been evaluated for their applicability to blend estimation of individual tobacco rods. Finally, development is in progress of a neural network to predict the likelihood of switching as a function of smoker demographics and brand attributes.

In the Cigarette Testing area effort is continuing to obtain ISO Guide 25 accreditation for the Consolidated CTSD/Product Audit Laboratory. An x-ray fluorescence method for determining the elemental composition of cigarette papers is under development.

As part of the R&D Process Manufacturing effort, a recommendation has been made to purchase a moisture monitor for monitoring moisture at the entrance to and exit of the C-dryer for the Cast Leaf process. The evaluation of an at-line sugar monitor to test for uniformity of Burley Spray application is underway. Feasibility studies have been completed on several approaches to developing and implementing an on-line monitor for menthol-on-filler. An FTIR spectrometer has been obtained to evaluate as a potential rapid method for tobacco blend analysis.

Under Predictive Maintenance, a GD packer sub-assembly has been installed in the vibration laboratory. This is part of the effort to develop vibration analysis as a tool for predicting and diagnosing machine health for packers and other manufacturing equipment.

In the Print Web Inspection area, a plan will be presented to PM-Richmond management for the implementation of global inspection of print web on the printing press.

With regards to Research on Analytical Methods, the capability to apply high pressure liquid chromatography (HPLC)-Fast Atom Bombardment (FAB) mass spectrometry (MS) to the analysis of components in mixtures has been demonstrated.

A more detailed discussion of the above topics follow this summary.

PROGRAM TITLE: Biochemical Special Investigations

WRITTEN BY : O. M. Gaines

PERIOD COVERED: Third Quarter, 1992.

Objective

Develop biosensor technology in-house to allow the determination of the concentration of gas phase compounds of interest.

Status

Experiments were conducted to examine the interaction of an antibody with nicotine in gas phase. In order to shorten the exposure time to nicotine in gas phase the antibody was purified. A frequency shift was observed using the crystal coated with the purified antibody. Additional work has shown that the crystals can be cycled through at least 5 exposure-measurement-desorption phases per day while in the monitor. The crystals also retained their binding activity when tested on two successive days.

An experiment was conducted that demonstrated that lipid films can be applied to piezoelectric quartz crystals (10MHz) in an organic solvent. The crystals can be regenerated by the removal and deposition of a new lipid film.

Several experiments were performed to examine the interaction of lipid versus non-lipid coated crystals with gas phase synthetic menthol. Menthol was detected by phoshatidyl choline coated crystals at a sensitivity level that was 10 fold higher as compared to uncoated crystals. Crystals coated with 1-oleoyl-2-acyl, dioleoyl glycerol, and distearoyl glycerol detected menthol. Crystals coated with 1-oleoyl-2-acyl were the most sensitive to the presence of menthol whereas disteracyl-coated crystals were the least sensitive to the presence of menthol.

Plans

Continue efforts to determine if the antibody exposed to nicotine is a specific reaction. Measure the concentration of nicotine being exposed to antibody coated crystals.

Contributors

E. Morrisette, M. Steele and B. Davies.

Objective

Develop or implement a nicotine ELISA for the determination of nicotine concentrations in samples of interest to PM.

Status

The commercially available nicotine ELISA was used to quantitate nicotine synthase activity in tobacco root extracts required by the Tobacco Biochemistry Program. A set of five samples were tested in the nicotine ELISA. The assay detected less than 1 ppb nicotine in three of the control samples tested - dibasic sodium phosphate, nicotinic acid and enzyme preparation. The concentration of nicotine detected in two samples - a combination of buffer, nicotinic acid, N-

methylpyrrolinium and PD-10 purified enzyme; and a combination of buffer, nicotinic acid, N-methylpyrrolinium, and non-purified enzyme - was 5.4 and 102 ppb, respectively. Relative to their respective controls, no increase in nicotine was detected in either the crude or partially purified enzymatic preparations. However the data showed that PD-10 purification reduced the endogenous level of nicotine prior to experiments designed to measure the activity of nicotine synthase.

Plans

No further quantitation of nicotine synthase activity is planned at this time due to a restructuring of the Tobacco Biochemistry Program.

Contributors

M. Steele and E. Morrisette.

Objective

Determine if the Electronic Nose developed by the University of Warwick can be applied to several sensory and analytical questions currently being examined in PM laboratories.

Status

Samples of both acceptable and unacceptable commercially supplied glycerol samples were sent to the Electronic Nose group at Warwick. The samples were analyzed by tin oxide and polymer based sensors. These sensors were selected because of their successful application to the differentiation of coffees based on the head space odor. The sensors were optimized to detect low molecular weight volatile acids and aldehydes.

The tin oxide sensors differentiated 30% of the malodorous glycerol samples while the polymer sensors detected and differentiated only one of the malodorous samples. Analysis of the data indicated that the individual sensors used in each system were not optimal for this study.

Additional experiments were conducted at Warwick during a visit by a member of Project 6902. Fresh glycerol samples were supplied and used in the experiments which involved the use of a different protocoll Procedures were implemented to increase the concentration of the volatiles exposed to the sensors. During these experiments the tin oxide sensors clearly differentiated two unacceptable from two acceptable glyceroll samples. The members of the Electronic Nose group explained that the major malodorous odors organoleptically detected in the fresh malodorous glycerol samples mainly included amines, compounds for which the sensors were not optimized.

Plans

Discontinue the research effort with Warwick University. Develop and implement the use of a tin oxide sensor to conduct similar studies in-house.

202313513:

J. Lewis, S. Capocelli, B. Davies and M. Steele.

PROGRAM TITLE: Cigarette Testing Services WRITTEN BY

J. M. Garman, P. Grantham

PERIOD COVERED: Third Quarter, 1992

Objective - ISO 25 Accreditation

To pursue and obtain ISO Guide 25 accreditation for the consolidated CTSD/Product Audit Laboratory.

Status

The ISO 25 team has defined the scope of accreditation which identifies those analyses for which accreditation will be pursued. The team has assembled currently available documentation for those analyses. A project team was chartered to develop appropriate documentation for the circumference analysis. For each analysis and for the overall division, documentation covering the methods plus procedures and practices need to be updated and/or generated. The team has started drafting a quality manual for the division and has met with representatives of Allied-Signal's Hopewell facility to discuss ISO Guide 25. Allied-Signal's fibers and thermoplastic QC laboratories have been ISO 25 accredited since 1987.

Plans

Continue to draft a quality manual for the consolidated CTSD/Product Audit testing operation. Coordinate the development of appropriate documentation as outlined in ISO Guide 25.

Objective - Elemental Composition of Competitive Paper

To develop a routine Energy Dispersive X-ray Fluorescence method for determining the elemental composition of cigarette papers and include this information in competitive product reports.

Status

The X-ray fluorescence spectrometer was installed in May. Prior to its arrival, twenty cigarette papers from the marketplace and development were selected as possible standards for the method! These papers included both flax and wood pulp, different basis weights and a wide range of elemental additives. The papers were submitted to ARD for elemental analyses. Based on the information from ARD, ten papers were selected as standards. These standards were used to set up a method which quantitatively determines the concentration of Mg, P, K, Ca, and Ti in cigarette papers which are removed from rods. Initial runs of unknown papers from the Domestic and Asian CI have been analyzed using this method. Comparison with ARD analyses of the same papers are ongoing. Preliminary data looks very good. We are awaiting the final set of data from ARD, at which time a statistical analysis of the data will be performed.

Plans

Complete the statistical analysis of the data. Analyze all available CI samples for two production months. Determine how best to present the data in the CI reports. Complete the documentation of the method. The data should be ready for inclusion into the CI reports no later than October.

PROGRAM TITLE: Research on Analytical Methods

WRITTEN BY : N. Jensen

PERIOD COVERED: Third Quarter, 1992.

I. Objective

To initiate, develop and apply high performance liquid chromatography (HPLC)-fast atom-bombardment (FAB) mass spectrometry (MS) to the analysis of components in complex mixtures. (Contributor: T. Sumpter)

<u>Status</u>

HPLC-FAB MS capability has been established using the JEOL four-sector tandem mass spectrometer. This represents a significant enhancement of the current analytical capability of PM R&D. We have demonstrated the following attributes in real analysis:

(a) Analyses using analytical (ca. 4 mm i.d.), narrow bore, microbore, and capillary columns. (b) Analyses using post-column addition of FAB matrix which enables the use of any FAB matrix with any column/mobile phase combination except concentrated salts. (c) Analyses can be performed in either the positive or negative mode and either scanning or selected ion monitoring (SIM) may be used which offers advantages in selectivity. (d) Analyses have been applied to small polar, labile molecules (mw <200) and large molecules (mw >1000). The high performance capabilities of both LC and MS are best utilized with capillary HPLC-FAB MS. Rapid scan speeds allow detection of narrow peaks, and sensitivity has been demonstrated in the low femtomol range using peptides. Other projects undertaken include the analysis of flavor compounds, synthetic organic reaction products and intermediates, and sugars on tobacco filler.

Plans

Develop: collaborative interactions throughout R&D applying HPLC-FAB MS to the structural identification of components in mixtures.

II. Objective Introduce new analytical procedures and equipment. (Contributor: B. Ryan)

Status

A TN-05 nitrogen analyzer purchased from Cosa Instrument Corporation was installed in the Chemical Analysis Section and analysts trained in operation and maintenance. The unit is designed to measure total nitrogen in aqueous matrices by converting the chemically bound nitrogen to NO by high temperature vaporization/oxidation of the sample. The evolved NO subsequently reacts with ozone to form excited NO which is detected by chemiluminescence.

The R&D Methods Manual Committee reviewed the following methods at its June 29, 1992 meeting: Water in Tobacco and Tobacco Products and Anethole in Cigarettes and Cigarette Filler by HPLC. The two methods with the committee comments and revisions have been returned to the authors.

<u>Plans</u>

To apply these methods per customer requests.

PROGRAM TITLE: Process Monitoring WRITTEN BY: R. W. Kanipe

PERIOD COVERED: Third Quarter, 1992

I. Objective

To evaluate commercially available moisture monitors; to select and implement the appropriate monitors at the entrance to and exit of the C-dryer for the Cast Leaf Process. (M. Parrish and G. Hicks-White)

Status

A recommendation was made for purchase of the Moisture Systems Microquad 8000 with two sensors for installation at the entrance to and exit of the C-dryer. This recommendation was made after a thorough evaluation of several different technologies. The Microquad sensors have been installed, and a coarse calibration has been performed using both GC water and OV as the reference methods.

Plans

To complete the calibration and continue to provide technical support as required.

II. Objective

To evaluate an at-line sugar monitor to test for uniformity of burley spray application. (M. Parrish):

Status

Specialized software was purchased from Infrared Engineering for the TM55 sugar monitor which will compute the average and standard deviation of sugar measurements taken across a leaf. The standard deviation will be used as an indicator of the uniformity of burley spray application. The results of a baseline experiment showed no significant difference in the monitor response for the top and bottom of the leaf. The results of a second experiment indicate that the monitor can distinguish between cased and uncased material provided sufficient measurements are taken on a given strip of burley to adequately characterize the sugar monitor response.

Plans

To have James Gear assess the results of these studies to determine if the information is sufficient to calculate the number of strips that would have to be examined to compare burley spray application uniformity for conventional versus New Primary processing.

III. Objective

To develop and implement an on-line monitor for menthol-on-filler. (W. Lewis)

Status

Feasibility studies were completed! Results are summarized below:

- Scanning Near Infrared Reflectance (NIR) (Perstorp Analytical and LT Industries) Both vendors demonstrated limited correlation with menthol and the NIR response at selected wavelengths.
- 2. Short Wave Near Infrared Transmission (NIT) (Katrina) Limited correlation with menthol and the short wave NIR response at selected wavelengths was demonstrated.
- 3. NMR (Process Control Technology) No correlation could be made with the NMR spectra and menthol/level.
- 4. Supercritical Fluid Extraction (E. Thomas) Recoveries using this technique did not exceed 90% for a one hour extraction.
- 5. Thermal Treatment (W. Lewis) Menthol was generated in the headspace of thermally treated tobacco; quantitative recovery has not been determined.

Plans

To pursue development of the Katrina short wave NIT and the thermal treatment approaches.

IV. Objective

To develop and implement a rapid method for tobacco blend analysis. (C. Harward)

Status

Per S. Clark's request, the short term goals for this project were redefined to address the more immediate need for a rapid, single-rod blend analysis. An Analect Diamond 20 FTIR spectrometer was purchased for this method. This instrument has a spectral range that includes portions of the near and mid infrared regions. The procedure for sample preparation of cigarette filler has been determined. Because of the optical density of the material, diffuse reflectance techniques were employed to enhance the signal to noise ratio. A motor and sample assembly were fabricated to slowly rotate the sample while acquiring spectra. Thus, spectra are collected across a larger surface area. Analysis of the blended samples from the Bokelman Set is in progress.

To complete the IR analysis of the calibration set and evaluate the results. To coordinate with the General Analytical Section for XRF analysis on this same sample set.

V. Objective

To provide support to the NET moisture monitors.

Status

The TM55 moisture sensors were calibrated off-line using a three component expanded blend (65% bright, 20% MT, and 15% C34 burley) cased with 1.5% sugar/glycerin casing. The results showed that this calibration is also applicable to a 100% expanded bright material, similarly cased. The sensors are ready for installation on the NET process.

Plans

To provide support for calibration adjustment following installation on-line.

VI. Objective

To evaluate the reference method used for the calibration of on-line moisture monitors. (G. Hicks-White and B. Kanipe)

Status

As spectroscopic based monitors are measuring water, the premise is that much of the blend sensitivity can be removed by calibrating against a water specific method (GC) as opposed to oven volatiles. Operational plans have been written to utilize Semiworks as a test facility for this evaluation. The primary process was divided into the nine major locations of the moisture monitors. Each location has been assigned a phase number for the program. Phase I, entrance and exit to the pre-blend cylinder, has begun. A Microquad 8000 and two sensors were acquired (on-loan) from Moisture Systems. Off-line calibration (against OV and GC water) has been performed using bright, burley, and RL strip, and oriental leaf. The results of this calibration indicate that a combination curve can be fitted for bright, oriental, and RL when calibrated against GC water. For OV, the slopes for the individual materials were not different, but the intercept of RL compared to bright and oriental was significant as would be expected because of the presence of the other volatiles in RL. The burley responses were unusual when compared to the other materials and could not be included in a composite calibration curve using either OV on GC water.

Plans

To add RCB to the calibration set to ensure there are no unusual results with this material and to test DBC bright strip equilibrated to several moisture levels against the current

composite calibration. To collaborate with E. Baldwin of Moisture Systems in evaluating the results for burley.

VII. Objective

To determine the moisture and time at which non-damaged burley, oriental, and selected sheet materials (RCB and RLTC) develop visible mold. (G. Hicks-White)

Status

The laboratory studies have been completed, and the results have been reported in a presentation to QE and Leaf. The burley study has also been summarized in a memo.

Plans

To complete documentation of the oriental/sheet lab study. To complete a report combining the bright, burley, oriental and sheet studies and evaluate the relationships between the results of the three studies.

PROGRAM TITLE:

Computing Systems

WRITTEN BY

M. Allred, J. Blankinship, B. Kane, P. Lipford, J. Palesis

PERIOD COVERED:

Third Quarter, 1992

Objective - Semiworks - HP1000 Replacement

Replace the Semiworks HP 1000 system with a DEC Micro VAX system. Provide production and engineering reporting functions that equal or exceed those found in the HP 1000 system.

Status

The HP1000 computer has been replaced. Disposal of this equipment is in progress.

The new system consists of a VAX 3100 computer running the VMS operating system. Fortran programs capture Fisher-Provox run-time data and store them in RS/1 tables. RS/1 procedures written in RPL gather the data for a run, perform calculations and generate reports and graphs.

A Digital printer that is compatible with the Fisher-Provox, RS/1, and the VAX spooling facility was installed.

The system has run in the production mode for over two months without requiring significant software changes. A memo-documenting the completion of this project was distributed.

Plans

Provide support as needed!

Objective - CTSD - TPM Station Upgrade

Upgrade the CTSD TPM Workstations. Replace outdated equipment with current, more flexible equipment. Modify procedures to enhance throughput and accuracy.

Status

Two UNIX-based manual weighing stations (pm151 and pm152) are fully operational. They provide for initial and final weighing, accepting puff counts, editing data and reporting. These stations were upgraded to support user control of printer node and error reporting by date or by group run barcode:

The robotic weighing station is fully operational. The robot/balance control program allows selection of either or both of the balances and checks the light screen. Communications protocols between workstation and robot controller have been stable for two months.

The puff count program, which automatically collects puff counts from the robotic smoking machines, is fully operational.

Tasks that remain to be done involve downloading cigarette length information from the host database, elimininating the need to query the database for barcode ID and adjustments to code to manage information flow by Tray Number rather than barcode ID:

Plans

Turn the system over to CTSD the second half of September for final testing.

Objective - CTSD - PT Lab Upgrade

Provide the Physical Testing Lab with workstations for Cigarette Length, RTD, Filter Length, Circumference, Tobacco Weight, Filter Weight and Porosity.

Status

Six instruments (Filter Length, Cigarette Length, Circumference, Tobacco Weight, Filter Weight, Porosity) in this lab have been migrated from the obsolescent, inflexible Data Acquisition Systems (DAS's, blue boxes) to PC- based systems. The seventh conversion (RTD) is ready for final testing,

Plans

Complete testing of RTD and close the project.

Objective CTSD - Materials Evaluation Lab (MEL)

Automate data transfer between MEL instruments and the CTSD database.

Status

Two Firmness stations are in final testing. They are networked to the UNIX host. Procedures for uploading results to the host database are ready.

Two computers attached to balances in the D-building Sieve Room were networked to the UNIX host using PC NFS.

Three computers attached to the MEL CV/OV instruments were upgraded to 286 CPUs with hard disk and were networked to the UNIX host.

Plans

Integrate sieve room and CV/OV results into a host database to complete this project.

Objective - Reconstituted Tobacco - Castleaf

Capture information from three Castleaf lab instruments, transfer the information to the UNIX host and integrate it into a host database.

Status

A Logical Connection programmable switch was installed to connect three serial devices (Thwig-Albert tensile tester, balance, and micrometer) and printer to the PC in the lab. Programming specifications have been developed and program development is in progress.

Plans

Complete programming of data capture and transfer program. Define database load procedure.

Objective - Analytical Research - Lotus Macro for Tracor X-ray

Automate the computations and reporting for the Tracor X-ray.

Status

A Lotus macro was generated to process 10 - 15 raw data sample sets from the Tracor X-ray, check for differences in Calcium and Potassium, produce a warning message if the difference is greater than 3 and produce a report of calculated values and warning messages for each sample set.

Plans:

This project is complete. Provide support as needed.

Objective - Desktop Support

Provide planning, technical support, and integration for the desktop computing devices in use within the Research Center.

Status

The software/hardware audit effort of the R&D PC continues. The initial audit is complete. Significant work has been done with the database group to develop a System Management Database that will house and maintain the hardware and software configuration of each R&D computer system. The database design and implementation is complete. Loading the information gathered during the audit is about to begin.

The PC backup project is being evaluated. The initial plans have been temporarily been put on hold to evaluate solutions that best fit with our long term data management scheme.

Macintosh network access to New York has been requested by PED. An interium solution giving this access and meeting R&D security restrictions has been recommended, tested and implementation has begun. Due to the security restrictions the network speeds are somewhat degraded, but all requested applications have been tested and shown to work.

Finalize the PC backup strategy and begin implementation.

Complete implementation of the Macintosh, New York access project.

Initiate the 800-number software support of PC and Macintosh systems that was purchased with the DEC maintenance contract.

Objective - Installation/Transition to DECsystem 5900

Provide economies of scale and common availability of server hosts services for R&D computing requirements. Provide and manage shared computer resources. Integrate the heterogeneous computing environment to minimize inter-vendor incompatibilities.

Status

Phase III of the DECsystem 5900 Installation/Transistion Plan (provide vendor and system software suites comparable to the existing Vax/8650 systems) will be completed on August 31, 1992 and the Vax/8650 systems will be shutdown. Two interactive sessions were conducted in the R&D Lexture hall to present an overview of the transition to the DECsystem 5900 and to answer user questions and concerns. The physical de-installation of the two Vax/8650 systems will be completed by September 15, 1992. Implementation of Phase III is currently in progress. The remaining Vax/8650 system will be reconfigured and continue to be available to the user community with a code name of PM700.

Two 8mm tapes drives on the Vax/8650 systems have been de-installed and re-installed on the DECsystem 5900 system. Unattended system backup software has been ported and implemented on the DECsystem 5900 utilizing the 8 millimeter tape technology.

Plans

Complete Phase III of the DECsystem 5900 Installation/Transition Plan.

Objective - BL Dryer Belt Tracking

Use expert systems, fuzzy logic, and machine learning to improve tracking of the BL Dryer Belt and thus substantially increase production. Improved tracking will increase production by:
(1) minimizing downtime, (2) minimizing wear and tear of critical process components, and (3) making it possible to expand the width of the belt which carries the slurry through the dryer.

Status

(a) Tracking improvements: Development of the expert system has continued on the BL dryer belt of Line II. Presently, the expert system is performing dramatically better than the PID controller on both head and tail ends of the belt. Based on tracking data obtained on August 21, the expert system has reduced the tail end oscillation range from 0.68 inches to 0.37 inches (a 46 percent improvement over PID control) and the head end oscillation range from 0.94 inches to 0.61 inches (a 35 percent improvement over PID control).

- (b) Immediate Benefits: A study conducted by the BL Plant Management demonstrated that the tracking improvements under expert system control can save the plant an estimated \$265,000 per year by reducing downtime (SAVAGE, J., 'Tracking Related Downtime Costs', Inter-office memo to Larry Murphy, July 2, 1992). In response to a request by the BL Plant, the current developmental version of the expert system is being used on a 24 hour basis for Line II tracking. BL production line operators and supervisors have been trained to run the expert system.
- (c) Feasibility of belt widening: In order to widen the BL dryer belts from 59 inches to 61.4 inches, the stop switch will need to be positioned at 0.75 inches from either edge of the belt. Thus, tracking with the wider belt should be maintained within +/-0.5 inches to be reliable. Results obtained from expert system tracking with the 59 inch belt on Line II (continuous tracking for ten days) are as follows: (1) under normal operational conditions, the belt is maintained within +/-0.35 inches; (2) under minor disturbance conditions (such as belt scraping and belt cleaning), the belt is maintained within +/-0.4 inches; (3) under extreme disturbance conditions (such as sheet recasting and major adjustment of the rubber roller pressure) belt tracking exceeds +/-0.5 inches but is less than +/-0.75 inches; (4) during belt startup, tracking exceeds +/-1.4 inches due to the dramatic temperature changes affecting the dynamics of the belt. Thus, these results indicate that the present version of the expert system would efficiently handle steady-state tracking of the 61.4 inch belt under normal operational conditions and under minor disturbance conditions. Further improvements are needed to enable the expert system to handle efficiently the tracking of the 61.4 inch belt under severe disturbance conditions and during startup.

Improve expert system performance in order to maintain belt tracking within +/-0.5 inches under severe disturbance conditions and under startup conditions.

Objective - Statistical Analysis of Chromatographic Data

Determine the statistically significant and important differences between gas chromatograms of sidestream smoke for control and test cigarettes. Employing a variety of multivariate analysis and pattern classification techniques, investigate the physical and chemical meaning of these differences.

Status

Chromatographic data were statistically analyzed from a series of sidestream chamber runs for control and test cigarettes coated with CR-2978. Both raw and normalized patterns were analyzed using descriptive and inferential statistics. Based on an analysis of variance of the normalized data, 112 compounds were identified as significantly different between the control and CR-2978 models. Importantly, these differences were virtually all in one direction: all but three of the 112 compounds were higher in the CR-2978 model than in the control. This result is also consistent with the finding that the total area of the chromatograms is significantly higher for the CR-2978 models than for the control. A report was issued documenting the principal results of this study.

Plans for further chromatographic analyses by D. Douglas or F. Hsu are unclear at this point. In the meantime, chemometric techniques in factor analysis, partial least squares, and pattern recognition will be studied in preparation for further work in this area.

Objective - Blend Analysis

Develop techniques to estimate, classify, and recognize the composition of a tobacco blend given a set of observed analytical and spectroscopic properties. Also, determine which analytical properties provide the most information about blend composition.

Status

Several types of multivariate calibration techniques were evaluated for their applicability to blend estimation of individual tobacco rods - including multiple linear regression, principal component regression, partial least squares (PLS) regression, linear mixture models, and neural networks. The two most promising techniques appear to be (1) PLS "forward" regression and (2) linear mixture "reverse" maximum likelihood estimation. A research report is being written which summarizes these calibration techniques.

Plans

Complete research report on multivariate calibration techniques. Develop PLS software for blend estimation of individual tobacco rods.

Objective - Neural Network Model of Brand Switching

Develop neural network and other predictive models which can be used to predict brand switching behavior as a function of smoker demographics and brand attributes.

Status

As the first stage in this modeling project, a set of neural network models were developed to predict the attributes of the post-switch brand as a function of smoker demographics and the attributes of the pre-switch brand. The explanatory variables for the models are the demographics (region, age, race, sex, education, income, and marital status) of the smoker and the attributes (length, menthol/non-menthol, packing, tipping color, price, and tar level) of the pre-switch brand. Except for age which is a continuous variable, all the inputs to the neural network are binary variables coded using a 1-of-n representation. There are a total of 28 inputs to the neural network. The training outputs are the corresponding attributes of the post-switch brand, coded as 1-of-n binary variables. After training, the predicted outputs are continuous variables between zero and one which represent the likelihood or market share of the associated post-switch attribute. A separate neural network was developed for each post-switch attribute - length, menthol/non-menthol, packing, tipping color, price, and tar level. A

training set of 8727 examples was constructed from the 1990-91 survey data in the "Tracking" study, and includes only whites who have switched brands during the past two years. These particular models do not predict the likelihood of brand switching per se - but rather, for those smokers who do switch, the attributes of the products into which they switch.

In the course of model development, several enhancements to the backpropagation training methodology were identified and implemented to improve the learning speed and generalization capabilities of the neural network model. A decision support system was also developed for the delivery of the neural network models to the Product Evaluation Division.

Work has started on the second stage of model development. A training set of over 60000 examples was constructed from the 1990-91 Tracking data for the development of a neural network model to predict the probability of switching. The explanatory variables for this model are the demographics (region, age, race, sex, Nielsen County, education, income, and marital status) of the smoker, the attributes of the smoker's brand, and possibly brand family. The output is the predicted probability of switching within a two-year time frame.

Plans

Complete development of neural network to predict the likelihood of switching. Determine whether including the pre-switch brand family as an explanatory variable improves the predictive performance of the model.

Develop neural network models to predict the attributes of the post-switch brand as a function of smoker demographics; the attributes of the pre-switch brand, and possibly the pre-switch brand family. Determine whether including the pre-switch brand family as an explanatory variable improves the predictive performance of the model.

Develop neural network models to predict the post-switch brand family as a function of smoker demographics, the attributes of the pre-switch brand, and the pre-switch brand family.

Using the above models, develop a decision support system to predict brand switching/cannibalism for new products—i.e. what types of smokers of what types of products will switch to a new product. This can assist in the development and marketing of new products most likely to increase P.M. sales.

Develop a neural network model to predict the probability of quitting as a function of smoker demographics, the attributes of the smoker's brand, and possibly brand family.

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PROGRAM TITLE: Print Web Inspection

WRITTEN BY : K. H. Shafer

PERIOD COVERED: Third Quarter, 1992

Objective

Support QA and Purchasing in the implementation of global inspection of print web on the printing press.

Status

The 100% inspection of web on a press at J. W. Fergusson with the Futec system showed earlier this year that gross defects which have in the past caused product to be put on hold can be detected at the press during the printing of the web. We had planned to assess the reliability of the system over a longer period of time under normal operating conditions at the printer. This effort involves renting a system from Futec for \$60,000 to \$80,000 with intent to purchase for \$300,000 and finding a press dedicated to the production of PM product so that competitors' products are not inspected. A vendor has not been found who is willing to purchase such a system or who has a dedicated press.

Furthermore, information is unavailable which adequately details the financial impact on PM. An alternative strategy is to inform our vendors through an information package of the defect detection capability of the Futec system emphasizing through the Purchasing Department that such defects are to be eliminated from our production stream. Concurrent with this action would be the implementation of a system by QA to better track the costs associated with incoming materials defects

Plans

A plan will be presented to Ken Houghton, Don Knudson, and Bill Moore by Janice Busic and Chris Irving.

PROGRAM TITLE: Predictive Maintenance

WRITTEN BY : K. H. Shafer

PERIOD COVERED: Third Quarter, 1992

Objective

Develop vibration analysis as a tool for predicting and diagnosing machine health and as a design aid for improving machine performance.

Status

A GD packer sub-assembly (second wheel) has been installed in the vibration laboratory. The sub-assembly has both Geneva and cam-follower mechanisms which are used to generate indexing and reciprocating motions in packers. A DC motor and a speed controller were connected to drive the sub-assembly. Initial measurements indicated a misalignment between the motor shaft and the sub-assembly drive. The shaft was aligned so as not to contribute to the vibration of the sub-assembly.

Sensors have been received for monitoring the performance of the main drive including the left knee of five packers located in the A-line of Bay-1 at the Manufacturing Center. The optimum sensor locations and mounting procedure are being determined.

The universal mounting system for the mechanical shaker system was tested. Overall system performance was adequate. A design modification was required to optimize the performance. The new design will also include a modification that will extend the shaker capability for exciting a structure in the horizontal plane.

Plans

Develop strategies for the design of monitoring and diagnostic techniques for packer operations. Install and operate packer sub-assembly to obtain vibration signals. Determine the ideal locations for mounting accelerometers on the main drive and 1st wheel of five packers in Bay-II. Obtain viscoelastic damping links from 3MI Company and test on a lab scale system designed to investigate machine dynamics modifications.

Source: https://www.industrydocuments.ucsf.edu/docs/ltjl0000